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| 10/041,558 | 01/10/2002 | Wolfram Burst | 52097 | 2747 |
| 26474 | 7590 08/31/2006 | | EXAMINER | |
| NOVAK DRUCE DELUCA & QUIGG, LLP | | | SINGH, PREM C | |
| 1300 EYE STREET NW SUITE 400 EAST TOWER | | | ART UNIT | PAPER NUMBER |
| WASHINGTON, DC 20005 | | | 1764 | |
| | | | DATE MAILED: 08/31/2006 | 5 |

Please find below and/or attached an Office communication concerning this application or proceeding.

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| | Application No. | Applicant(s) | | | | |
|---|---|-----------------------------|--|--|--|--|
| Office Action Summany | 10/041,558 | BURST ET AL. | | | | |
| Office Action Summary | Examiner | Art Unit | | | | |
| | Prem C. Singh | 1764 | | | | |
| The MAILING DATE of this communication appears on the cover sheet with the correspondence address Period for Reply | | | | | | |
| A SHORTENED STATUTORY PERIOD FOR REPLY IS SET TO EXPIRE 3 MONTH(S) OR THIRTY (30) DAYS, WHICHEVER IS LONGER, FROM THE MAILING DATE OF THIS COMMUNICATION. - Extensions of time may be available under the provisions of 37 CFR 1.136(a). In no event, however, may a reply be timely filed after SIX (6) MONTHS from the mailling date of this communication. - If NO period for rèply is specified above, the maximum statutory period will apply and will expire SIX (6) MONTHS from the mailing date of this communication. - Failure to reply within the set or extended period for reply will, by statute, cause the application to become ABANDONED (35 U.S.C. § 133). Any reply received by the Office later than three months after the mailing date of this communication, even if timely filed, may reduce any earned patent term adjustment. See 37 CFR 1.704(b). | | | | | | |
| Status | | | | | | |
| 1)⊠ Responsive to communication(s) filed on 02 Au | <u>ugust 2006</u> . | | | | | |
| 2a)⊠ This action is FINAL . 2b)☐ This | action is non-final. | | | | | |
| 3) Since this application is in condition for allowar | Since this application is in condition for allowance except for formal matters, prosecution as to the merits is | | | | | |
| closed in accordance with the practice under E | closed in accordance with the practice under Ex parte Quayle, 1935 C.D. 11, 453 O.G. 213. | | | | | |
| Disposition of Claims | | | | | | |
| 4)⊠ Claim(s) 1,3 and 6-15 is/are pending in the app | olication. | | | | | |
| 4a) Of the above claim(s) is/are withdrawn from consideration. | | | | | | |
| 5) Claim(s) is/are allowed. | | | | | | |
| 6)⊠ Claim(s) <u>1,3 and 6-15</u> is/are rejected. | | | | | | |
| 7) Claim(s) is/are objected to. | | | | | | |
| 8) Claim(s) are subject to restriction and/or | r election requirement. | | | | | |
| Application Papers | | • | | | | |
| 9) The specification is objected to by the Examine | r. | | | | | |
| 10)⊠ The drawing(s) filed on <u>10 January 2002</u> is/are: a)⊠ accepted or b)□ objected to by the Examiner. | | | | | | |
| Applicant may not request that any objection to the drawing(s) be held in abeyance. See 37 CFR 1.85(a). | | | | | | |
| Replacement drawing sheet(s) including the correction is required if the drawing(s) is objected to. See 37 CFR 1.121(d). | | | | | | |
| 11) The oath or declaration is objected to by the Examiner. Note the attached Office Action or form PTO-152. | | | | | | |
| Priority under 35 U.S.C. § 119 | | | | | | |
| - | main aith condon 25 LL C C S 110/a |) (d) or (f) | | | | |
| 12) Acknowledgment is made of a claim for foreign | priority under 35 U.S.C. § 119(a) |)-(a) or (1). | | | | |
| a) ⊠ All b) □ Some * c) □ None of: | s have been received | | | | | |
| 1. Certified copies of the priority documents | | on No | | | | |
| 2. Certified copies of the priority documents | • • | | | | | |
| 3. Copies of the certified copies of the prior | • | ed III tilis National Stage | | | | |
| application from the International Bureau (PCT Rule 17.2(a)). | | | | | | |
| * See the attached detailed Office action for a list of the certified copies not received. | | | | | | |
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| Attachment(c) | | | | | | |
| Attachment(s) 1) Notice of References Cited (PTO-892) 4) Interview Summary (PTO-413) | | | | | | |
| 2) Notice of Draftsperson's Patent Drawing Review (PTO-948) Paper No(s)/Mail Date. | | | | | | |
| 3) Information Disclosure Statement(s) (PTO-1449 or PTO/SB/08) Paper No(s)/Mail Date 5) Notice of Informal Patent Application (PTO-152) 6) Other: | | | | | | |
| Paper No(s)/Mail Date 6) Other: | | | | | | |

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DETAILED ACTION

Claim Rejections - 35 USC § 102

The following is a quotation of the appropriate paragraphs of 35 U.S.C. 102 that form the basis for the rejections under this section made in this Office action:

A person shall be entitled to a patent unless -

(b) the invention was patented or described in a printed publication in this or a foreign country or in public use or on sale in this country, more than one year prior to the date of application for patent in the United States.

Claims 1, 6, 9, 10, and 13 are rejected under 35 U.S.C. 102(b) as being anticipated by Perry (Chemical Engineers Handbook by Perry and Chilton, Fifth Edition, McGraw Hill, 1973, Page 13-39).

Applicant's invention per claim 1 is about a method of separating a liquid mixture which forms at least one azeotrope by azeotropic distillation with an entrainer which additionally forms a binary and ternary azeotrope with the other components to be separated. The azeotropes have boiling points lower than that of entrainer. The azeotropes are separated into two parts: A, H containing fraction depleted in B and B, H containing fraction depleted in A. At least a part of the auxiliary H is introduced at the top and/or in the upper region of a column for distillation.

Perry reference (Figure 13-44) teaches schematically an azeotropic distillation process similar to the one claimed by the applicant's claim 1 in which benzene is added to separate ethanol-water azeotrope. The overhead product from the column which

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produces ethanol as a bottom product is a vapor with a composition near the ternary azeotrope formed by these three components. Table 13-12 lists water-ethanol-benzene as a ternary azeotropic mixture (Page 13-41). Minimum boiling point azeotropic mixtures water-ethanol, water-benzene, and ethanol-benzene are shown in Table 13-10.

It is to be noted that Perry's ethanol, water, and benzene are representing components A, B, and H respectively, of the applicant.

Perry's figure **13**-44 shows that the azeotropes are separated in to two parts: aqueous phase and ethanol phase. Aqueous phase is rich in water and depleted in ethanol, while the ethanol phase is rich in ethanol and depleted in water. Perry invention further teaches that it seems best to introduce the solvent in the top section of the column (Page **13**-42 first paragraph).

Perry's reference discloses the boiling points of binary azeotropes as follows: water-ethanol: 78.15°C, water-benzene: 69.25°C, ethanol-benzene: 68.24°C (Table 13-10). It is to be noted that benzene (entrainer) has a boiling point of 80.1°C.

Claim 6 of the applicant says that the auxiliary entrainer is obtained as bottom product and is re-circulated at least partly to the top or into the upper region of the column.

Perry reference teaches in figure **13**-44 that benzene-rich stream from column (C) is mixed with the original feed and taken into the upper portion of the column (A).

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Claim 9 of the applicant says that binary azeotrope AH and/or BH is a heteroazeotrope.

Perry reference shows in Table **13**-10 that the system: water-benzene forms two phases, which means the system forms a heteroazeotrope.

Applicant's claim 10 discusses that the liquid or liquefied AH containing and/or BH containing fraction is subjected to a phase separation to give A-rich or B-rich phase and an H-rich phase is returned to the column.

Perry's figure **13**-44 shows a separator (B) that separates the components in two layers: the top layer with 14.5% ethanol, 84.5% benzene, and 1% water; while the bottom layer with 53% ethanol, 11% benzene, and 36% water. The figure further shows that the benzene-rich phase is returned to the column (A).

Claim 13 of the applicant mentions a process wherein the liquefied n-butanol/water fraction is subjected to a phase separation to give n-butanol-rich phase and a water-rich phase and the n-butanol-rich phase is separated by distillation into a fraction enriched in n-butanol and a fraction depleted in n-butanol.

Perry reference mentions in figure **13**-38 that liquefied n-butanol/water feed enters a decanter (phase separator), which operates at a temperature below the boiling point. The butanol-rich phase from the decanter is fed to a stripping column which produces high purity alcohol as the bottom product and an overhead vapor which approaches the azeotropic composition. The aqueous phase is fed to a second stripper

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which produces butanol-free water as a bottom product. Since water is the bottom product, open steam can be used to provide "reboil" vapor. The aqueous column also produces a top vapor which approaches the azeotropic composition. Both overhead vapor streams are condensed in a common condenser and then fed to the decanter along with the fresh feed (Page **13**-37 column 2, and Page **13**-38 Column 1).

Claim Rejections - 35 USC § 103

The following is a quotation of 35 U.S.C. 103(a) which forms the basis for all obviousness rejections set forth in this Office action:

(a) A patent may not be obtained though the invention is not identically disclosed or described as set forth in section 102 of this title, if the differences between the subject matter sought to be patented and the prior art are such that the subject matter as a whole would have been obvious at the time the invention was made to a person having ordinary skill in the art to which said subject matter pertains. Patentability shall not be negatived by the manner in which the invention was made.

The factual inquiries set forth in *Graham* v. *John Deere Co.*, 383 U.S. 1, 148 USPQ 459 (1966), that are applied for establishing a background for determining obviousness under 35 U.S.C. 103(a) are summarized as follows:

- 1. Determining the scope and contents of the prior art.
- 2. Ascertaining the differences between the prior art and the claims at issue.
- 3. Resolving the level of ordinary skill in the pertinent art.
- 4. Considering objective evidence present in the application indicating obviousness or nonobviousness.

Claims 3, 7, 8, 11, and 15 are rejected under 35 U.S.C. 103(a) as being unpatentable over Perry (Chemical Engineers Handbook by Perry and Chilton, Fifth Edition, McGraw Hill, 1973, Page13-39).

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Claim 3 of the applicant says that the mixture to be separated is introduced continuously into the column.

Perry reference teaches that the azeotropic separations are used more frequently in batch separations (Page **13**-41, column 2, paragraph 1).

It would have been obvious to one skilled in the art at the time the invention was made to modify Perry invention and conduct the azeotropic separation continuously for a large scale operation and better control of the process.

Claim 7 of the applicant says that A, H containing fraction is taken off at a point above the feed point and B, H containing fraction is taken off at a point below the feed point.

Perry's figure **13**-44 discloses that ethanol, benzene, and a small amount of water are taken from the top of the column (A) and water is taken out from the bottom of column (D).

It would have been obvious to one skilled in the art at the time the invention was made to modify Perry invention and combine the operations of columns (A) and (D) and take out water from the bottom of column (A) to make the overall separation process more economical.

Claim 8 of the applicant mentions that the mass flow of auxiliary entrainer introduced is from 0.5 to 15 times the mass flow of the mixture to be separated.

Perry reference teaches an azeotropic system: benzene-cyclohexane-acetone (acetone being the solvent) in figure 13-42. The acetone/fresh feed ratio must be such as to give a total feed which falls on the straight material-balance line between the two end products (Page 13-41, column 1, paragraph 4). Although it is a different system than ethanol-water-benzene, the principle of solvent/feed ratio applicable to both systems should be the same.

It would have been obvious to one skilled in the art at the time the invention was made to modify Perry invention and specify the solvent/feed ratio to ensure that the total feed falls on the straight material-balance line between products (A) and (B).

The applicant mentions in claim 11 that component A is selected from the group consisting of chlorinated hydrocarbons and monocyclic C_6 - C_{10} aromatics and component B is selected from the group consisting of C_3 - C_8 alkanols and the auxiliary H is water.

Perry reference provides in Table 13-10 minimum boiling point azeotropic binary mixture of tetrachloroethylene (also called as perchloroethylene or PERC) (A) and butanol (B). The invention does not specifically mention about the entrainer.

It would have been obvious to one skilled in the art at the time the invention was made to modify the teachings of Perry as disclosed in figure 13-44 by substituting PERC in place of ethanol (A), butanol in place of water (B) and water in place of benzene (H) and conduct the process. The use of an entrainer causes better and easier separation.

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The applicant in claim 15 mentions that the auxiliary H further comprises a base.

Perry reference does not mention about a base in the entrainer.

It would have been obvious to one skilled in the art at the time the invention was made to add a base in the entrainer. Using a base mixed with water will reduce the corrosive action of the chlorinated hydrocarbons and increase the life of the equipment.

Claims 12 and 14 are rejected under 35 U.S.C. 103(a) as being unpatentable over Perry in view of Ohe (Vapor-Liquid Equilibrium Data by S. Ohe, Elsevier Science Publishers, Amsterdam, 1999, #758 and #763,764).

Claim 12 of the applicant mentions that the chlorinated hydrocarbon is perchloroethylene and the alkanol is n-butanol and the mixture optionally further comprises butyl chloride.

Perry reference does not mention about butyl chloride.

Standard texts and Ohe present data on relative volatility and vapor-liquid equilibrium of perchloroethylene-n-butanol and butyl chloride-n-butanol azeotropic systems.

It would have been obvious to one skilled in the art at the time the invention was made to combine the teachings of Perry and Ohe to make optional use of butyl chloride along with perchloroethylene to use a blend of two chlorinated hydrocarbons instead of one to give more flexibility to the process.

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The applicant in claim 14 mentions that a fraction which comprises butyl chloride and water and is largely free of perchloroethylene and butanol is additionally obtained as lowest boiling fraction.

As mentioned under claim 12, standard texts and Ohe present azeotropic distillation data for the system butyl chloride-n-butanol-water.

It would have been obvious to one skilled in the art at the time the invention was made to combine the teachings of Perry and Ohe to show that butyl chloride-water could be obtained as the lowest boiling fraction. The reason as explained under claim 12, was to achieve more flexibility for the process.

Response to Arguments

Claim rejections under 35 USC §102

The Applicant argues that A, B, and H form two binary azeotropes, AH and BH. In addition thereto, H is theoretically capable of forming a ternary azeotrope with A and B. Also, in the process of instant claim 1, the AH and BH containing fractions are isolated; that is pure A or pure B are not formed. In contrast, Perry discloses the isolation of pure B (ethanol) and the formation of a ternary azeotrope.

The Applicant's argument is not persuasive because Perry discloses two binary azeotropes (Table **13**-10, page **13**-38): AH (ethanol-benzene) and BH (water-benzene). Also, benzene is capable of forming a ternary azeotrope with water and ethanol. Also, Perry isolates AH and BH. According to the claim 1, AH fraction depleted in B, and BH

fraction depleted in A does not exclude AH fraction with zero B or BH fraction with zero A.

Claim rejections under 35 USC §103

The Applicant argues that there is no teaching, suggestion or motivation to modify Perry to arrive at the claimed invention. It is desirous to avoid the formation of the ternary azeotrope ABH. This is accomplished by introduction at least a part of the auxiliary H at the top and/or upper region of a column, as described in the Applicant's examples. While Perry describes introducing benzene via a top region of a distillation column, Perry nonetheless, discloses the formation of ternary azeotropes.

The Applicant's argument is not persuasive because Perry passes the auxiliary H in the top region of the column and cautions, "In most cases, it seems best to introduce the solvent in the top section of the column, but such generalizations are dangerous.

The availability of computers makes a thorough investigation of feed- and solvent-introduction points possible." (Page **13**-42, column 1, paragraph 1)

The Applicant argues that the claimed invention requires fewer separation columns than Perry.

The Applicant's argument is not persuasive because Perry produces 100% pure product. One skilled in the art could reduce the number of separation columns at the cost of product purity.

The Applicant argues with regard to claim 11 that one having ordinary skill in the art would not have recognized from Perry that the use of water as auxiliary H did not lead to the formation of a ternary azeotrope.

The Applicant's argument is not persuasive because the choice of an auxiliary H (entrainer) is not random since Perry discloses as an example with benzene-cyclohexane binary system, "The first step in the search for a good solvent is to compile a list (from Horsley's tabulation) of all substances which form an azeotrope with either benzene or cyclohexane or both. The new azeotrope(s) should boil at least 10°C above or below the benzene-cyclohexane azeotrope to make separation easy in the azeotropic distillation column." (Page 13-41, column 1, paragraph 4).

Conclusion

Applicant's amendment necessitated the new ground(s) of rejection presented in this Office action. Accordingly, **THIS ACTION IS MADE FINAL**. See MPEP § 706.07(a). Applicant is reminded of the extension of time policy as set forth in 37 CFR 1.136(a).

A shortened statutory period for reply to this final action is set to expire THREE MONTHS from the mailing date of this action. In the event a first reply is filed within TWO MONTHS of the mailing date of this final action and the advisory action is not mailed until after the end of the THREE-MONTH shortened statutory period, then the shortened statutory period will expire on the date the advisory action is mailed, and any extension fee pursuant to 37 CFR 1.136(a) will be calculated from the mailing date of

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the advisory action. In no event, however, will the statutory period for reply expire later than SIX MONTHS from the date of this final action.

Any inquiry concerning this communication or earlier communications from the examiner should be directed to Prem C. Singh whose telephone number is 571-272-6381. The examiner can normally be reached on MF 6:30 AM-3:00 PM.

If attempts to reach the examiner by telephone are unsuccessful, the examiner's supervisor, Glenn Caldarola can be reached on 571-272-1444. The fax phone number for the organization where this application or proceeding is assigned is 571-273-8300.

Information regarding the status of an application may be obtained from the Patent Application Information Retrieval (PAIR) system. Status information for published applications may be obtained from either Private PAIR or Public PAIR. Status information for unpublished applications is available through Private PAIR only. For more information about the PAIR system, see http://pair-direct.uspto.gov. Should you have questions on access to the Private PAIR system, contact the Electronic Business Center (EBC) at 866-217-9197 (toll-free).

ps/081806

Glenn Caldarola Supervisory Patent Examiner Technology Center 1700